Optimization and Characterization of Bio-Diesel Production from Rubber Seed Oil Using Response Surface Methodology

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Abstract: The global pursuit of decarbonization and net-zero emissions necessitates sustainable and renewable energy alternatives, with biodiesel emerging as a viable solution. This study focuses on utilizing rubber seed oil (RSO), a non-edible and abundant feedstock, for biodiesel production. RSO addresses the dual challenge of reducing reliance on fossil fuels and minimizing competition with food resources. However, the high free fatty acid (FFA) content of RSO presents a significant barrier to efficient biodiesel synthesis through traditional alkaline transesterification. A two-step process, acid esterification followed by alkaline transesterification, was employed to overcome this limitation. The process was optimized using Box-Behnken Design (BBD) within the Response Surface Methodology (RSM) framework. The ideal conditions for maximum biodiesel yield included a methanol-to-oil molar ratio of 5:1, catalyst concentration of 1.19%, reaction time of 79 minutes, and reaction temperature of 61.5°C. Under these parameters, a maximum biodiesel yield of 97.09% was achieved. The resultant biodiesel, rubber seed oil methyl ester (RSOME), was characterized in accordance with ASTM and EN standards. RSOME exhibited superior thermal stability, high flash and fire points, and notable calorific properties, making it a competitive alternative to conventional diesel fuel. By valorizing an underutilized waste

resource, this study not only contributes to the advancement of sustainable biodiesel production but also aligns with global decarbonization initiatives. The findings underscore the potential of RSO-derived biodiesel as a critical component of future renewable energy strategies, supporting environmental sustainability and economic viability.

1. Introduction

The role of fossil fuels in accelerating climate change is both significant and multifaceted. Fossil fuel combustion remains the dominant source of anthropogenic greenhouse gas emissions, especially carbon dioxide (CO2), which heavily contributes to atmospheric imbalances and global warming. This ongoing emission disrupts the carbon cycle, intensifies climate change, and results in a variety of environmental and health challenges. Recent research indicates a continuous upward trend in emissions stemming from fossil fuel usage and cement production, stressing the need for a swift transition to renewable energy alternatives (1,2). Such alternatives, including biodiesel derived from rubber seeds, offer innovative solutions to these challenges. Given the environmental and health risks associated with fossil fuels, there is a pressing need for policies and strategies that support sustainable energy practices to mitigate their adverse effects. The global carbon budget offers compelling evidence of the overwhelming influence of fossil fuels on climate change. Current data shows that fossil fuels contribute over 75% of greenhouse gas emissions globally, with nearly 90% of CO2 emissions directly attributable to their combustion. Precise tracking and analysis of these emissions are vital for guiding the development of sustainable alternatives such as rubber seed biodiesel (2). Biofuel production can substantially reduce carbon footprints and support global net-zero ambitions (3,4). Combustion processes release particulate matter and other pollutants into the air, significantly impacting public health. Studies estimate that air pollution from fossil fuel emissions is responsible for millions of premature deaths annually, particularly in densely populated and industrialized areas (5). Furthermore, the environmental effects are equally dire, as fossil fuel combustion contributes to rising sea levels, extreme weather events, and other climate anomalies, which exacerbate vulnerabilities in already marginalized communities. Rubber seeds, a largely underutilized resource found abundantly in tropical regions, are an ideal feedstock for biodiesel production. Research demonstrates that high-quality biodiesel produced from rubber seed oil (RSO) possesses properties comparable to conventional diesel, making it a viable alternative (1).The production process of rubber seed biodiesel involves critical steps such as saponification, acidification, and esterification. These processes refine the oil, reducing viscosity and enhancing energy efficiency, thereby ensuring the biodiesel's suitability for widespread use (2). Catalyst selection is one of the most crucial factors in optimizing biodiesel production processes. Studies have shown that using limestone as a catalyst achieves high conversion efficiencies of up to 96.9%, while ensuring that the biodiesel meets ASTM D6751 standards for quality (5). Furthermore, the recyclability of catalysts enhances the sustainability and cost-effectiveness of biodiesel production. Another important consideration is the free fatty acid (FFA) content of rubber seed oil, which requires controlled processing conditions for optimal results. Preheating rubber seeds and managing their storage conditions are essential for reducing moisture and FFA levels, improving biodiesel yields (3,4). Comparative research on various biodiesel feedstocks, including soybean and flaxseed oils, has identified key production factors such as alcohol-to-oil ratios, reaction temperatures, and durations that can significantly enhance biodiesel yields (6,7). Under optimized conditions, biodiesel yields from soybean and flaxseed oils have exceeded 90%, demonstrating the potential for similar successes with rubber seed oil. For rubber seed biodiesel to achieve widespread

adoption, supportive policy frameworks are essential. Eliminating fossil fuel subsidies, which distort energy markets and favor polluting fuels, is a crucial step in creating a level playing field for biofuels (8). Moreover, policies promoting research and development in advanced biofuel technologies can enhance production efficiency and reduce associated costs. Collaborative initiatives that involve scientists, policymakers, and local communities ensure that biodiesel projects align with climate goals while delivering tangible socioeconomic benefits (9). Rubber seed biodiesel offers a practical solution for reducing emissions, especially in sectors where electrification remains challenging, such as heavy-duty transportation and aviation (4,10).

This study explores its exclusive focus on rubber seed oil (RSO) as a biodiesel feedstock, emphasizing its renewable, sustainable, and non-edible nature. It employs a tailored two-step transesterification process to address the high free fatty acid content of RSO, ensuring optimal conversion to biodiesel. Furthermore, the study utilizes advanced optimization techniques, specifically Response Surface Methodology (RSM) with Box-Behnken Design, to refine reaction parameters and maximize biodiesel yield. Unlike broader studies, this research rigorously characterizes the biodiesel properties against ASTM and EN standards, ensuring its practical applicability and alignment with environmental sustainability goals

2. Methodology

The methodology employed in this study focuses on a comprehensive and systematic approach to biodiesel production using rubber seed oil, a non-edible feedstock with a high free fatty acid (FFA) content. This methodology is designed to address the limitations of conventional alkali-catalyzed transesterification, which often results in soap formation when used with high-FFA oils, thereby reducing yield and complicating downstream processes. To overcome these challenges, a two-step transesterification process is utilized, combining acid esterification to reduce FFA content and alkaline transesterification to convert triglycerides into biodiesel (Fig.1). This integrated approach ensures high conversion efficiency and yields biodiesel that meets stringent international fuel standards.

Fig.1:Experimental Setup

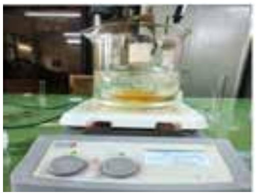


Fig.2:Stirring Setup View

Fig.3:Seperation Funnel

Fig.4: Flash Point Tester

The production process begins with the pre-treatment of raw rubber seed oil, which is collected, filtered, and heated to 60°C to remove impurities and reduce viscosity, facilitating smooth filtration. Following pre-treatment, the physicochemical properties of the oil are analyzed. The initial FFA content is determined to be 15.98%, necessitating an acid esterification step to reduce it to an acceptable level for alkali-catalyzed transesterification. The density of the oil is measured as 0.925 g/cm^3, while its calorific value is determined to be 38.5 MJ/kg, indicating its potential as an energy-rich biodiesel feedstock.

The second step, alkaline transesterification, is designed to convert the triglycerides in the esterified oil into biodiesel. Potassium hydroxide (KOH) is used as the base catalyst, while

methanol serves as the reactant. The reaction is carried out in a 500 mL three-neck conical flask equipped with a condenser, magnetic stirrer, and hot plate to maintain consistent reaction conditions. The temperature is controlled between 55°C and 65°C, and the mixture is stirred continuously at 700 rpm to ensure uniform mixing and efficient interaction between the reactants (Fig.2). The reaction proceeds until the triglycerides are fully converted into fatty acid methyl esters. After completion, the reaction mixture is transferred to a separating funnel and allowed to settle overnight, resulting in the formation of two distinct layers: a yellow biodiesel layer on top and a black glycerol layer at the bottom (Fig.3). The glycerol is carefully drained off, leaving the crude biodiesel for further purification. The purification of biodiesel is a crucial step to remove residual contaminants such as unreacted methanol, catalysts, soap, and other impurities. The crude biodiesel is washed multiple times with lukewarm water to ensure thorough removal of these residues. The washed biodiesel is then subjected to a drying process, where it is heated to evaporate any remaining water or volatile compounds. Finally, the biodiesel is filtered through fine filter paper to remove any remaining particulates, resulting in a clean, high-quality product. The purified biodiesel is analyzed to confirm its compliance with fuel standards. Efficient esterification achieved at time 60 minutes, temperature 61°, stirring speed at 720 rpm (Table 1).

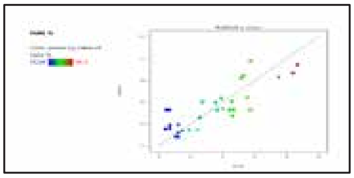
The optimization of process parameters is performed using Response Surface Methodology (RSM) with a Box-Behnken design, which evaluates the effects of critical factors such as methanol-to-oil molar ratio, catalyst concentration, reaction temperature, and reaction time on biodiesel yield (Table 2). A total of 30 experimental runs are conducted based on this design, and the results are analyzed using Design-Expert software to develop a quadratic regression model that describes the relationship between these variables and biodiesel yield. Analysis of Variance (ANOVA) confirms the statistical significance of the model, with regression coefficients (R^2) exceeding 0.95, indicating strong predictive accuracy. Fit statistics such as Root Mean Square Error (RMSE) and Mean Absolute Error (MAE) are minimized to values below 0.02 and 0.01, respectively, demonstrating the precision and reliability of the model. The optimized conditions derived from the model predict biodiesel yields exceeding 92%, which are experimentally validated, achieving yields ranging from 91% to 94%. Post-production, the biodiesel is subjected to rigorous quality assessments to ensure compliance with ASTM D6751 and EN 14214 standards. Key fuel properties such as flash point, calorific value, and viscosity are measured. The flash and fire points are determined using a Pensky-Martens closed cup tester, where the biodiesel is gradually heated, and the temperature at which it emits ignitable vapors is recorded. The calorific value, indicative of the energy content of the biodiesel, is measured using a bomb calorimeter (Fig.4), while viscosity is assessed using a viscometer to confirm compatibility with engine requirements.

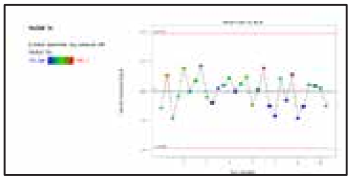
Table 1: Data of Esterification Process

Methanol to oil ratio	Weight of oil	Weight of methanol (gm)	Weight percent-age of H2SO4 (wt/wt oil)	Weight of H2SO4 (gm)	Yield (%)	Acid - Value (mg of NaOH/gm of oil)
6:1	30	5	1.2%	0.36	97.0926%	1.12

Table 2: Independent Variables and levels used for Response Surface Design

Factors	Name	Units	Low	High
A	Methanol to oil ratio (w/w% of oil)	% of oil	5	7
B	Amount of Catalyst (w/w% of oil)	% of oil	0.3	1.2
C	Reaction Time	minutes	55	90
D	Reaction Temperature	C	52	62





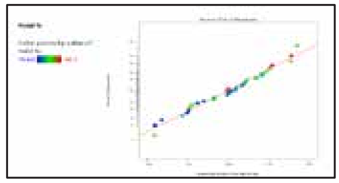


Fig.5.1: Yield%;Predicted vs Actual Fig.5.2:Yield%; Residuals vs Run Fig.5.3: Yield%; Plot of Residuals

The optimized methanol-to-oil molar ratio, catalyst concentration, reaction temperature, and time ensure maximum yield and efficiency (Fig.5). The measured properties of the biodiesel, including density, calorific value, and flash point, validate its compliance with industrial fuel standards. This methodology, integrating pre-treatment, acid esterification, alkaline transesterification, and optimization through RSM, demonstrates a robust and scalable approach to biodiesel production. By addressing the challenges of high FFA content and utilizing advanced statistical techniques for optimization, the study provides a comprehensive framework for producing biodiesel from rubber seed oil, contributing to the development of sustainable and renewable energy solutions.

3. Result

The analysis involved determining key biodiesel properties, including dynamic viscosity, kinematic viscosity, and specific gravity. The density of the biodiesel was calculated to be 813.3 kg/m³, and its dynamic viscosity was measured at 24.4 cP. From this, the kinematic viscosity was derived as 30.00 mm²/s, and the specific gravity was found to be 0.8133. These properties are essential for evaluating the fuel's compliance with standard biodiesel specifications and its performance in combustion applications.

The calorific value of the biodiesel was also assessed to determine its energy content, yielding a higher calorific value of 46.97 MJ/kg. This value indicates the energy efficiency of the biodiesel and confirms its suitability as a renewable alternative to conventional fossil fuels.

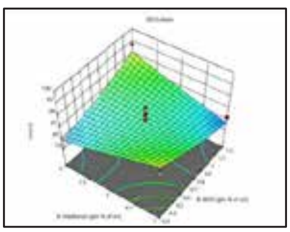


Fig.6.1: Methanol-Oil Ratio Catalyst

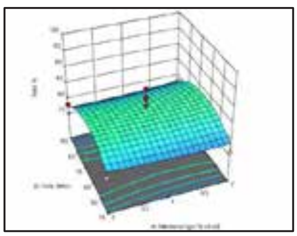


Fig.6.2: Methanol-Oil Ratio Temperature

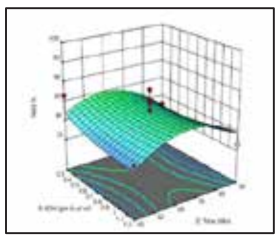


Fig.6.3: Catalyst Temperature

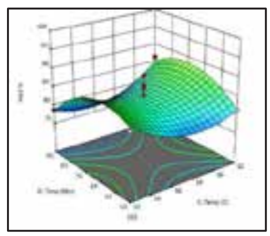


Fig.6.4: Reaction Time-Temperature for esterification yield

To further optimize the production process, a 3D response surface methodology (RSM) analysis was conducted to examine the combined effects of process parameters, including methanol concentration (wt/wt % of oil), catalyst amount (wt/wt % of oil), reaction time, and reaction temperature. The 3D plots highlighted the relationships between these variables and their influence on biodiesel yield (Fig.6). The interaction between two independent variables was analyzed to understand their combined impact on conversion rates. The highest point on the response surface represented the optimal conditions for maximizing biodiesel yield, offering insights into the most effective parameter combinations for producing Rubber Seed Oil Methyl Ester (RSOME)

4. Conclusion

The research successfully produced biodiesel from rubber seed oil (RSO) using a two-step esterification and transesterification process, achieving a maximum yield of 97.09% under optimal conditions identified through Response Surface Methodology (RSM). The ideal parameters included a methanol-to-oil molar ratio of 5:1, catalyst concentration of 1.19% (wt/wt of oil), reaction temperature of 61.5°C, and reaction time of 79 minutes. The produced biodiesel, Rubber Seed Oil Methyl Ester (RSOME), was thoroughly characterized to ensure compliance with ASTM and EN standards. These results highlight RSOME's viability as a sustainable alternative to conventional diesel, contributing significantly to green energy initiatives.

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